
वायु प्रदूषण को मापने की पद्धति
भाग 26 निकल

Method For Measurement
of Air Pollution

Part 26 Nickel

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FOREWORD

This Indian Standard (Part 26) was adopted by the Bureau of Indian Standards, after the draft finalized by the Air Quality Sectional Committee had been approved by the Chemical Division Council.

Air pollution is a challenging environmental problem faced by modern society. Knowledge of the concentration of potentially toxic substances, like heavy metals, in the airborne particulate is essential for assessing the pollution levels and providing important information for evaluating the level of long term exposure to the population.

Nickel is well known as a human carcinogen. Occupational exposure to nickel has been found to correlate with an increase in the frequencies of prostate, skin, esophageal, nasal and lung cancers in particular. Nickel is liberated in ambient air mainly from metallurgical plants. Therefore, developing an authentic method for its measurement is very important.

In the formulation of this standard, considerable assistance has been derived from the following documents:

- a) USEPA Compendium method IO – 3.2;
- b) USEPA Compendium method IO – 3.5;
- c) Journal of Analytical Atomic Spectrometry; and
- d) NAAQS Guidelines for the measurement of ambient air pollutants – Volume I of CPCB.

The composition of the Committee responsible for the formulation of this standard is given in Annex A

In reporting the results of a test or analysis in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD FOR MEASUREMENT OF AIR POLLUTION PART 26 NICKEL

1 SCOPE

This standard prescribes the following methods for measurement of nickel in ambient air:

- a) Atomic Absorption Spectrometric (AAS) method.
- b) ICP-MS method.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (<i>third revision</i>)
4167 : 1980	Glossary of terms relating to air pollution (<i>first revision</i>)

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4167 and the following shall apply.

3.1 Particulate Nickel — That collected on EPM 2000 filter paper.

4 ATOMIC ABSORPTION SPECTROSCOPY (AAS)

4.1 Principle

This method is based on acid digestion and atomic absorption spectroscopy for the chemical analysis of nickel in ambient air samples collected on filter paper using a high volume sampler. If flame atomic absorption is used, the amount of deposited particulate nickel collected on the filter shall be greater than 1 µg. Determination by graphite furnace atomic absorption spectrometry allows measurement of quantities of less than 1 µg, but is only applicable after experimental validation of detection limits.

4.2 Detection Limit

The minimum detection limit of the method with flame atomic absorption spectrometry is 0.1 µg/ml. This corresponds to a concentration of 5 ng/m³ when

sampling is done for 8 h at an average flow rate of 1.1 m³/min in a sample solution of 25 ml.

4.3 Apparatus

4.3.1 High Volume Sampler

4.3.2 Hot Plate (*Thermostatically Controlled*)

4.3.2 Microwave Digestive system

4.3.4 Atomic Absorption Spectrophotometer (AAS) — Equipped with air/acetylene and nitrous oxide/acetylene burner heads or graphite furnace.

4.3.5 Analytical Balance

4.3.6 Filter Media — EPM 2000 of size 20.3 × 25.4 cm (8 × 10 inches).

4.4 Reagents

4.4.1 Concentrated Hydrochloric Acid (AR Grade) — see IS 265.

4.4.2 Concentrated Nitric Acid (AR Grade) — see IS 264.

4.4.3 Digestion Solution — Dilute 30 ml concentrated HNO₃ and 80 ml concentrated HCl to 1 000 ml with distilled water.

4.4.4 Stock Nickel solution (1 000 µg/ml) — Certified grade.

4.4.5 Intermediate Nickel Solution (100 µg/ml) — Dilute 10 ml of stock nickel solution (see 4.4.4) to 100 ml with distilled water.

4.4.6 Standard Nickel solution (10 µg/ml) — Dilute 10 ml of intermediate nickel solution (see 4.4.5) to 100 ml with distilled water.

4.4.7 Working Nickel Standards — Working nickel standard solutions (100 ml) preferably from 0.05 µg/ml to 1 µg/ml are prepared from the standard nickel solution (see 4.4.6) by taking appropriate volumes of it and diluting with distilled water.

4.5 Procedure

4.5.1 Sampling Procedure

Ambient air sample is collected on EPM 2000 glass micro-fibre filters by means of high volume sampler.

The specified duration of sampling is commonly 8 h or 24 h. During this period, several readings (hourly) of flow rate should be taken. After sampling period, calculate the volume of air sampled. Take out the filter media from the sampler and put in a container or envelope.

4.5.2 Sample Storage

During transport of filters to the laboratory, care should be taken to minimize contamination and loss of sample. The filters should be transported in a container or envelope with temperatures maintained upto 30 °C. The samples shall be analyzed within 180 days.

4.5.3 Extraction of Samples

The collected sample on glass micro-fibre filters may be extracted either by hot plate procedure or by microwave extraction method.

4.5.3.1 Microwave extraction

Cut the filter into pieces by means of a clean stainless steel scissor. Place the filter pieces in a labelled centrifuge tube using plastic forceps or vinyl gloves. Add 10 ml of digestion solution to each centrifuge tube. Using the plastic forceps crush the filter pieces so that the digestion solution covers all the pieces.

Place the centrifuge tubes in a teflon vessel containing 31 ml of distilled water. Place the vessel caps with the pressure release valves on the vessels hand-tight and tighten using the capping station to a constant torque of 12 ft-lb. Place the vessels in the microwave carousel. Connect each sample vessel to the overflow vessel using the Teflon connecting tubes. Place the carousel containing the 12 vessels on to the turntable of the microwave unit. Irradiate the sample vessels at 486 W (power output) for 23 min.

Allow the pressure to dissipate, then remove the carousel containing the vessels and cool in tap water for 10 min. Using the capping station, uncap the microwave vessels, remove the labelled centrifuge tubes. Add 10 ml of distilled water to each centrifuge tube. Cap the centrifuge tubes tightly and mix the contents thoroughly for 2-3 min to complete extraction. The final extraction volume is 20 ml based upon the above procedure. Filter the extracted fluid with Whatman No. 41 and make up the final volume to 100 ml, the filtered sample is now ready for analysis

4.5.3.2 Hot plate procedure

The filter is cut into pieces by means of a clean stainless steel scissor. The pieces are placed in a 250 ml beaker. Digestion solution is poured into the beaker so that it covers the filter pieces. The beaker is placed on a hot plate (temperature below 80°C), contained in a fume hood, and refluxed gently while covered with a watch glass for 30 min. The beaker contents shall not be allowed to dry up. The beaker is removed from the hot

plate and allowed to cool. The beaker walls are rinsed with distilled water. The digested sample is filtered into a 100 ml volumetric flask through a Whatmann No.1 filter paper and diluted to the mark with distilled water. The extracted sample is now ready for analysis.

4.5.4 Blank Sample

To prepare a blank, glass micro-fibre filter, that has not been used for sampling is used. It is treated using the same procedure as used for the filter on which sample has been collected (*see* either 4.5.2 or 4.5.3) and the extracted fluid is used as blank.

4.6 Calibration

Working nickel standard solutions (*see* 4.4.7) are prepared and at least three standards are selected to cover linear range. The standards are aspirated into the flame or injected into the furnace. The absorbances are recorded and the calibration graph is plotted using absorbance and concentration, in µg/ml. The wavelength for nickel is 232 nm.

4.7 Analysis

The samples are aspirated into the flame or injected into the furnace and the absorbances are recorded. The concentration, in µg/ml, is calculated using the calibration curve.

4.8 Calculation

The nickel content in ambient air in µg/m³ can be calculated as follows:

$$C = \frac{(M_s - M_b) \times V_s}{V}$$

where

C = concentration of nickel in ambient air in, µg/m³;

M_s = concentration of nickel in extracted sample, in µg/ml;

M_b = concentration of nickel in blank sample, in µg/ml;

V_s = total volume of digested sample = 100 ml ; and

V = volume of air sampled, in m³.

5 INDUCTIVELY COUPLED PLASMA – MASS SPECTROSCOPY (ICP-MS)

5.1 Principle

The ambient air is sampled using a high volume sampler. The collected sample in solution form is introduced by pneumatic nebulization into a radio frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a quadrupole mass spectrometer having

a minimum resolution capability of 1 amu peak width at 5 percent peak height. The ions transmitted through the quadrupole are registered by a continuous dynode electron multiplier or Faraday detector and the ion information processed by a data handling system.

5.2 Detection Limits

Instrument Detection limit = 0.004 µg/l

NOTE — The instrument detection limit taken from 'Standard methods for the examination of water and waste water, APHA, 21st Edition'

5.3 Interferences

Several interfering sources may causes inaccuracies in the determination of nickel by ICP/MS.

5.3.1 Isobaric Elemental Interferences

Isobaric elemental interference is caused by isotopes of different elements that form single or double – charged ions of the same nominal mass-to-charge ratio and cannot be resolved by mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric elemental interference. Of the analytical isotopes recommended for use with this method, only molybdenum –48 (ruthenium) and selenium – 82 (krypton) have isobaric elemental interferences.

5.3.2 Physical Interferences

Physical interferences are associated with the physical processes that govern the transport of sample into the plasma and the transmission of ions through the plasma – mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer, at the point of aerosol formation and transport to the plasma, or during excitation and ionization processes within the plasma itself.

5.3.3 Isobaric Polyatomic Ion Interferences

Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest and that cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components.

5.3.4 Abundance Sensitivity

Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The potential

for these interferences should be recognized and the spectrophotometer resolution adjusted to minimize them.

5.3.5 Memory Interferences

Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the sampler and skimmer cones and from the buildup of sample material in the plasma torch and spray chamber. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element should be estimated prior to analysis.

5.4 Apparatus

5.4.1 ICP/MS Instrument, capable of scanning the mass 5–250 amu with a minimum resolution capability of 1 amu peak width at 5% peak height.

5.4.2 Argon Gas Supply, high purity grade, 99.9 percent.

5.4.3 Variable Speed Peristaltic Pump, required for solution delivery to the nebulizer.

5.4.4 Mass Flow Controller, required on the nebulizer gas supply. A water cooled spray chamber may reduce some types of interferences.

5.4.5 Electron Multiplier Detector – If an electron multiplier detector is being used, precautions should be taken, where necessary to prevent exposure to high ion flux. Otherwise changes in instrument response or damage to the multiplier may result.

5.4.6 Hot Plate, capable of having surface temperature of 150 °C.

5.5 Reagents

5.5.1 Stock Nickel Solution (1 000 µg/ml Ni) – Dissolve 0.1 gm nickel powder in 5 ml concentrated. nitric acid, heating to dissolve. The nickel powder should have been dried for 1 h at 105 °C. The stock solution is stored in a teflon bottle. CRM grade/NMI traceable nickel Standard solution (1000 µg/ml) purchased commercially may also be used.

5.5.2 Standard Nickel Solution (10 µg/ml Ni), prepared by diluting 1 ml of stock nickel solution (see 5.5.1) to 100 ml with distilled water containing 1 percent (v/v) nitric acid.

5.5.3 Nitric Acid (HNO₃), concentrated, ultra pure (see IS 264)

5.5.4 Perchloric Acid (HClO₄), concentrated, ultra pure.

5.5.5 Ashing Acid – 4:1 (v/v) $\text{HNO}_3 + \text{HClO}_4$ — Prepared by mixing 4 volumes of concentrated. HNO_3 with 1 volume concentrated HClO_4 .

5.5.6 Dilution Acid — Add 50 ml ashing acid (see 5.5.5) to 600 ml water and dilute to 1 litre.

5.6 Procedure

5.6.1 Sampling and Sample Storage

As per 4.5.1 and 4.5.2.

5.6.2 Digestion of Filter Paper

Cut the EPM 2000 filter into pieces and put in a clean beaker. Take a blank filter, cut it and put it in another beaker. Add 5 ml ashing acid to each beaker. Cover with a watch glass. Let the beakers stand for 30 min at room temperature. Heat the beakers on hotplate at 120 °C until about 0.5 ml remains. Add 2 ml ashing acid and repeat the previous step. Repeat the process until the solution is clear. Remove watch glass and rinse the wall of the beakers with distilled water. Heat the beakers again at 150 °C and reduce the sample to near dryness. Dissolve the residue in 2 to 3 ml dilution acid. Transfer the solutions quantitatively to 25 ml volumetric flasks. Dilute to mark with dilution acid.

5.6.3 Determination of Nickel by ICP-MS

The digested solution prepared (see 5.6.2) should be transferred to 10 ml centrifuge tubes.

Initiate instrument operating configuration. Tune and calibrate the instrument for the analytes of interest. Establish instrument software run procedures for quantitative analysis, a minimum of three replicate integrations are required for data acquisition. Discard any integrations considered to be statistical outliers and use the average of the integrations for data reporting, monitor all masses that might affect data quality during the analytical run. Use the rinse blank to flush the system between samples. Allow sufficient time to remove traces of the previous sample or a minimum of 1 min. Aspirate the samples for 30 seconds prior to the collection of data.

Samples having concentration higher than the established linear dynamic range should be diluted into range and re-analyzed. First analyze the sample for trace elements protecting the detector from the high concentration elements, if necessary by selecting appropriate scanning windows. Then dilute the sample to determine the remaining elements.

5.7 Calculation

The air volume sampled, corrected to normal condition:

$$V_{std} = V_s \left[\frac{T_{std}}{T_m} \right] \left[\frac{P_{bar}}{P_{std}} \right]$$

where

V_{std} = volume of ambient air sampled at normal conditions, m^3 ;

V_s = volume of ambient air pulled through the sampler, m^3 ;

T_{std} = absolute temperature, 298 K (25° C);

T_m = average ambient temperature, K;

P_{bar} = barometric pressure during sampling measurement condition, mm Hg;

P_{std} = normal pressure, 760 mm Hg; and;

Nickel concentration in the air sample can then be calculated as follows:

$$C = \frac{V(C_s - C_b)}{V_{std}}$$

where

C = concentration, $\mu\text{g nickel} / \text{m}^3$;

V = digestion volume = 25 ml;

C_s = concentration of nickel determined from sample using ICP-MS, $\mu\text{g/ml}$;

C_b = concentration of nickel determined from blank using ICP-MS, $\mu\text{g/ml}$; and

V_{std} = standard air volume pulled through filter, m^3 (at 25 °C and 760 mm Hg).

For data values less than 10, use two significant figures to report element concentrations. For data values greater than or equal to 10, three significant figures.

ANNEX A*(Foreword)***COMMITTEE COMPOSITION**

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